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IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :

KOU HASEGAWA :

SERIAL NO: 09/995,613 :

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FOR: POLISHING METHOD

DECLARATION UNDER 37 C.F.R. § 1.132

ASSISTANT COMMISSIONER FOR PATENTS  
ALEXANDRIA VIRGINIA 22313-1450

SIR:

Now comes Kou Hasegawa, who deposes and states that

1. I am named as an inventor in the above-identified application.
2. I am a graduated of Osaka University, Graduate School of Science and received my Master in Macromolecular Science degree in the year 1985.
3. I have been employed by JSR Corporation since 1985, and I have been conducting research in the field of CMP materials for 5 years.
4. I am familiar with the prosecution history of U.S Application Serial No.09/995,613 and I have read and understood the contents of the U. S. Patent No.6,194,317(Kaisaki et al) which was cited by the Examiner against the claims of 09/995,613.

In order to compare the polishing performance of polishing pads obtained using polymer particles by suspension polymerization with the polishing performance of polishing pads of present invention, the following experiments were carried out by me or under my direct supervision and control.

[1] Preparation of aqueous dispersions (E)

Aqueous dispersion (E), having a matrix material and abrasive dispersed therein

The respective components shown in Table 1 were loaded at the respective proportions into a temperature-adjustable autoclave, equipped with a stirrer, and were made to react suspension polymerization for 4 hours at 65°C. As a result, a suspension was obtained in which

styrene copolymer having 37um of mean particle diameter was dispersed.

The mean particle diameter was measured using a laser particle size analysis system made by Otsuka Electronics Co., Ltd. (in the description that follows, the mean particle diameter was measured by the same method).

Table 1

Component	Amount (Parts by mass)
Styrene	30
Methyl methacrylate	25
Isooctyl acrylate	40
Acryc acid	5
Poly vinylalcohol	1
Sodium lauryl sulfate	0.3
Benzoylperoxide	0.8
Ion-exchanged water	900

The suspension that was obtained was adjusted to pH 8.5 by means of a 25% aqueous solution of potassium hydroxide. Thereafter, ion-exchanged water was added, and after stirring under room temperature using Three-One Motor, a ceria ( $\text{CeO}_2$ ) powder, having a mean particle diameter of 0.3um prior to processing, was loaded and further stirring at 1,500 rotations/minute was carried out for 3 minute to obtain an aqueous dispersion (E).

Furthermore, this preparation was carried out by the same method as example 1 in the present invention.

## [2] Solidification

The above-described aqueous dispersions (E) was spread thinly on a polyethylene film and made flake-like in form by leaving and drying for 48 hours under room temperature. The flakes thus obtained were then powdered using a mixer. Then using the respective powders, disk-shaped polishing pads [E] of 30cm diameter and 3mm thickness were obtained using a mold press.

Furthermore, this solidification was carried out by the same method as example 1 in the present invention.

## [Test Example 1]

### [1] Polishing of tungsten wafers

#### (1) The method of the test

An aqueous chemical mechanical polishing solution (I) was prepared by incorporating hydrogen peroxide to an amount of 2% by mass and malonic acid to an amount of 1% by mass in ion-exchanged

water. The abrasive-containing polishing pads [E], which were obtained in the manner described above, which does not contain the abrasive, was adhered onto the surface table of a polishing device (model "LM-15," made by Lapmaster STF Corp.). Then using the polishing pad, a 4cm-square tungsten wafer (trade name, "SKW-5," made by SKW Co., Ltd.) was polished while supplying aqueous solution (I) at a rate of 150ml per minute. In this process, the table rotation speed was set to 50rpm, the polishing pressure was set to 350g/cm<sup>2</sup>, and the polishing time was set to 2 minute intervals.

The surface resistance value of the tungsten layer was measured by the DC 4-terminal method using a resistivity measuring device (model "Σ-10," made by NPS Corp.), and using the equation (1) shown below, and the thickness of the tungsten layer was calculated from the ratio with respect to the resistivity of tungsten. Then from the thickness values of the tungsten layer before and after polishing, the removal rate was calculated using the equation (2) shown below.

$$\text{Thickness of tungsten layer} = \{ \text{Resistivity of tungsten } (\Omega/\text{cm}) \} / \{ \text{Resistance value } (\Omega/\text{cm}^2) \} \cdots (1)$$

$$\text{Removal rate (A/minute)} = (\text{Thickness of tungsten layer before polishing} - \text{Thickness of tungsten layer after polishing}) / \text{Polishing time} \cdots (2)$$

Next, with an above-mentioned tungsten wafer, where the pitch is 200um (line width: 100um, spacer width: 100um), the distance  $T_0$ , from the tungsten layer surface to the bottom surface of the spacer, is 10,000A, and the distance  $t$ , from the tungsten layer surface to the surface of the insulating layer is 15,000A, polishing of the wafer surface was performed until the distance  $t$  became 20% the original distance. After polishing, the distance  $T_1$ , from the tungsten layer surface to the bottom surface of the indented part after polishing that is formed at the location where the spacer was formed, was measured, and the value  $(T_1/T_0)$ , obtained by dividing  $T_1$  by  $T_0$ , was indicated as the degree of surface flatness in Table 2. A smaller value of this degree of surface flatness indicates that polishing of excellent surface flatness can be performed.  $T_0$  and  $T_1$  were measured using a fine profile measuring device (model "P-10," made by KLA-Tencor Corp.).

## (2) The result of the test

The results are shown in Table 2 as Comparative example 15.

The results in Table 2 show that against the polymer particle size of Comparative example 15 being 37um, the polymer particle size of Example 1 is 166nm and the removal rate and degree of flatness of Example 1 and Example 2 of present invention are more excellent than these of Comparative example

15.

Furthermore, Example 1 and Example 2 in Table 2 are respectively the same to be Example 1 and Example 2 described in document of the present application.

Table 2

	Comparative example 15	Example 1	Example 2
Polymer particle size	37um	166nm	
Polishing pad	Polishing pad [E] (containing abrasive)	Polishingpad [A] (containing abrasive)	Polishing pad [B] (containing abrasive)
Liquid used	aqueous solution (I) (containing no abrasive)	aqueous solution (I) (containing no abrasive)	aqueous solution (I) (containing no abrasive)
Removal rate (A/min)	990	1080	1250
Degree of flatness(T1/T0)	0.06	less than 0.01	less than 0.01

[Test Example 2]

[2] Polishing of wafers with copper layer

(1) The method of the test

Aqueous chemical mechanical polishing solution (III) was prepared by incorporated hydrogen peroxide of an amount of 4% by mass, quinaldic acid of an amount of 0.3% by mass, malonic acid of an amount of 1% by mass, and potassium dodecylbenzenesulfonate of an amount of 0.02% by mass in ion-exchanged water and adjusting the pH9.0 by ammonia.

The above-mentioned polishing pads [E] were adhered onto the surface table of the above-mentioned polishing device, and while supplying aqueous solution (III) at a rate of 150ml per minute, a 4cm-square wafer with copper layer was polished. In this process, the table rotation speed was set to 50rpm, the polishing pressure was set to 350g/cm<sup>2</sup>, and the polishing time was set to 2 minute intervals.

The removal rate of copper layer was measured in the same manner as described above.

Next, with the above-mentioned copper wafer, where the pitch is 200um (line width: 100um, spacer width: 100um), the distance T<sub>0</sub>, from the copper layer surface to the bottom surface of the spacer, is 7,000A, and the distance t, from the copper layer surface to the surface of the insulating layer is 15,000A, polishing of the wafer surface was performed until the distance t became 20% the original distance. After polishing, the distance T<sub>1</sub>, from the copper layer surface to the bottom surface of the indented part after polishing

that is formed at the location where the spacer was formed, was measured, and the degree of surface flatness, obtained in the same manner as described above using the value ( $T_1/T_0$ ), obtained by dividing  $T_1$  by  $T_0$ , was indicated as the degree of surface flatness in Table 3.

(2) The result of the test

The results are shown in Table 3 as Comparative example 16.

The results in Table 3 show that against the polymer particle size of Comparative example 15 being 37 $\mu$ m, the polymer particle size of Example 1 is 166nm and the removal rate and degree of flatness of Example 7 of present invention are more excellent than these of Comparative example 16.

Furthermore, Example 7 in Table 3 is the same to be Example 7 described in document of the present application

Table 3

	Comparative example 16	Example 7
Polymer particle size	37 $\mu$ m	166nm
Polishing pad	Polishing pad [E] (containing abrasive)	Polishing pad [A] (containing abrasive)
Liquid used	aqueous solution (III) (containing no abrasive)	aqueous solution (III) (containing no abrasive)
Removal rate (A/min)	3080	3200
Degree of flatness( $T_1/T_0$ )	0.04	less than 0.01

6. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

長谷川 亨  
 \_\_\_\_\_  
 Signature Kou Hasegawa  
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 September 5, 2003  
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 Date